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**(54) Method of collapsing preforms for optical fibers**

(57) The present invention provides a method of preparing preforms for optical fibers. The invention allows one to remove or significantly reduce undesirable refractive index variations in the central portion of the optical fibers. The method of preparing the preform having a central duct includes the steps of a first collapsing step, an etching step and a second collapsing step. The first collapsing step reduces the size of the central duct without closing the central duct by heating the preform at a first preform collapsing temperature. A portion of the last deposited layer of the core glass layers is etched by flowing an etchant gas through the central duct at a lower temperature than the preform collapsing temperature. The preform is finally collapsed at a second collapsing temperature to close the central duct of the preform and form a solid rod.

Case Name: M.S. Fee 7-4  
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## Description

### Field Of The Invention

[0001] The present invention relates to a method of preparing preforms for optical fibers. More particularly, the invention relates to a method to remove or significantly reduce undesirable refractive index variations in the central portion of the optical fibers.

### Background Of The Invention

[0002] One of the processes in manufacturing a glass optical fiber is to collapse a preform having a central duct into a solid glass rod. During the process of closing the central duct, volatile dopants, such as germanium, are desorbed or released from one location of the preform structure. These dopant molecules are then either redeposited at another location or transported out of the preform. Due to the redeposition and/or transportation of these dopants, an undesirable refractive index deviation at the center of the core is formed. Fig. 1 demonstrates a refractive index profile of a multimode glass optical fiber in the prior art. The refractive index deviation takes the form of spikes or dips. Such refractive index deviation is detrimental in high-speed systems with restricted launch conditions.

[0003] Hence, one has tried to solve this problem in the prior art. United States Patent 4,793,843 discloses a method of manufacturing an optical fiber preform. In the process described therein, a gaseous etchant consisting of a combination of oxygen and a fluorocarbon compound  $C_2F_6$  flows through a central duct of the preform when the preform is heated to collapse. That is, the etching occurs at the same time when the preform is collapsed. Hence, during the etching, the activated processes of fluorine diffusion into the glass structure and volatilization of germanium occur because the etching is done at collapsing temperatures. United States Patent 4,793,843 specifies that the central duct is approximately equal to one millimeter before the gaseous etchant is passed through the central duct. When the central duct size is small, the risk to cause the duct to close due to surface tension makes the process difficult to control. If the duct closes prematurely while forming the preform, it may cause the increase of the glass attenuation and undesirable airlines may be formed within the preform. Although the resulting solid preform and optical fibers drawn from the preform have an improved refractive index profile, United States Patent 4,793,843 is limited to the etching with  $C_2F_6$ . Moreover, it is specifically disclosed that etching with  $SF_6$  at collapsing temperatures is not as effective as etching with  $C_2F_6$  at the collapsing temperatures. In other prior art (e.g., United States Patent 5,761,366; United States Patent 4,557,561; and Schneider et al., Proceedings of ECOC 1982) etching with  $SF_6$  during collapse is disclosed, but not as a separate step from collapsing.

### Summary Of Invention

[0004] The present invention provides a method to remove or significantly reduce undesirable refractive index variations in the central portion of the optical fibers. A method of preparing a preform for an optical fiber according to the present invention comprises the following steps. A preform having a central duct is heated at a first collapsing temperature to reduce the size of the central duct. The surface of the central duct is then etched at a temperature lower than the minimum collapsing temperature of the preform to remove a portion of deposited core materials. Finally, the preform is heated again at a second collapsing temperature to collapse completely the central duct of the preform. The etching step is performed by flowing an etchant gas through the central duct. The etchant gas comprises a mixture of oxygen and  $SF_6$ . The temperature for etching is preferred to be about 200-400°C lower than the minimum collapsing temperature of the preform. The etching temperature is about 1600-2000°C in the invention.

[0005] In a first embodiment of the present invention, three collapsing passes are performed through a traversing torch in the direction from the preform intake to the preform exhaust at a temperature of  $2150 \pm 100^\circ C$  at decreasing torch traverse speeds in the first heating/partial collapsing step. The etching step is performed by flowing an etchant gas at about  $1800^\circ C$  through the central duct. The etchant gas comprises a mixture of 6 sccm of  $SF_6$  and 194 sccm of  $O_2$ . Then, the preform is finally collapsed at a temperature of  $2200 \pm 100^\circ C$ .

[0006] In a second embodiment of the present invention, three collapsing passes are performed through a traversing torch in the direction from the preform intake to the preform exhaust at a temperature of  $2250 \pm 150^\circ C$  at decreasing torch traverse speeds. The etching step is performed by flowing an etchant gas at about  $1800^\circ C$  through the central duct. The etchant gas comprises a mixture of 6 sccm of  $SF_6$  and 194 sccm of  $O_2$ . In the final collapsing step, the preform is collapsed at a temperature of  $2220 \pm 100^\circ C$ .

[0007] In a third embodiment of the present invention, three collapse passes are performed through a traversing torch in the direction from the preform exhaust end to the intake end while the exhaust end is plugged with a device to prevent gases from exiting the preform. Then, the device is removed from the exhaust end, and the etching step is performed by flowing an etchant gas of 6 sccm of  $SF_6$  and 194 sccm of  $O_2$  while the preform is heated at a temperature of  $1800 \pm 150^\circ C$ . Finally, the preform is collapsed at  $2200 \pm 100^\circ C$  to form a solid rod.

### Brief Description Of The Drawings

[0008] The features and elements of the present invention will be better understood from the following detailed description of preferred embodiments of the

invention in which:

Fig. 1 is a refractive index profile for a prior art multimode fiber prepared in accordance with a conventional method.

Fig. 2 is a refractive index profile for a multimode fiber prepared in accordance with the present invention.

Fig. 3 is a differential mode delay diagram for a prior art optical fiber.

Fig. 4 is a differential mode delay diagram for an optical fiber produced in accordance with the present invention.

#### **Detailed Description Of Preferred Embodiments**

[0009] The present invention discloses a method to prepare a preform without having substantial refractive index variations such as spikes, dips and/or plateaus at the center of refractive index profile. This goal is accomplished by partially collapsing the preform which has a central duct, etching a portion of the last deposited layer of the core at a temperature lower than a minimum collapsing temperature of the preform and finally collapsing the preform to form a solid rod. When an optical fiber is drawn from a preform prepared in accordance with the invention, the optical fiber exhibits a refractive index profile with greatly reduced central defects. When the inventive procedure is optimized, it produces a smooth refractive index in the center of the optical fiber.

[0010] According to one aspect of the invention, a mixture of oxygen and  $\text{SF}_6$  is used as an etchant gas during the etching step. This mixture of gases decomposes yielding a higher concentration of F atoms at lower temperatures than fluorinated hydrocarbons suggested in the prior art (United States Patent 4,793,843) because  $\text{SF}_6$  molecules have lower bond energies. Thus, the etching step in the invention occurs at 200-400°C lower than the minimum collapsing temperature. Moreover, because of the lower temperature etching, the present invention significantly reduces diffusion of fluorine into the glass and volatilization of germanium during the etching step. The etchant gas has a flow rate of 3.0-60.0 sccm for  $\text{SF}_6$  and 50-1500 sccm for oxygen. The etch rate during the etching step ranges from 0.003  $\text{cm}^3/\text{min.}$ -0.08  $\text{cm}^3/\text{min.}$

[0011] According to another aspect of the invention, the present invention allows a considerably larger airline size due to a lower processing temperature than those in the prior art. The mixture of gases decomposes at relatively low temperatures and allows etching on the central duct significantly larger than what is used in the prior art, thereby creating a significantly more robust process. The lower temperature reduces the diffusion length for fluorine into the glass matrix. Thus, the absolute size

of the airline is less critical than what is described in the prior art. An optical preform manufactured in accordance with the present invention does not have apparent central dips even when the central duct is as large as 5 mm. The etch temperature is approximately 1600-2000°C, preferably at 1800°C. The separation of the etching step at a lower temperature from the collapsing step at a higher temperature in the invention makes it easier to keep the central duct open during the etching step. This is a significant advantage when a large-scale production of the preforms is adopted.

[0012] The present invention is used to prepare both multimode and singlemode preforms manufactured by a modified chemical vapor deposition (MCVD) process (see, e.g., United States Patent 4,334,903). A MCVD preform without a center dip in the refractive index profile is prepared by the following steps:

1 Use MCVD process to deposit a barrier and core glass layers on the inner face of a quartz substrate tube to form a preform having a central duct;

2 Heat the preform to reduce the size of the central duct at a first collapsing temperature;

3 Flow an etchant gas through the central duct at a temperature about 200-400°C lower than a minimum collapsing temperature to etch a portion of the deposited core from the central duct of the preform; and

4 Completely collapse the central duct of the preform at a second collapsing temperature.

[0013] The present invention is now explained below with a detailed description of preferred embodiments of the present invention in association with figures. Note that collapsing temperatures are defined as temperatures which result in the slow viscous flow of molten glass towards the center of the tube, driven by forces due to surface tension, causing a reduction in the tube diameter.

#### **Embodiment 1**

[0014] A graded index MCVD multimode preform is manufactured from a Heraeus Synthetic Quartz substrate tube. Glass layers for the core are deposited in the inner face of the tube through the MCVD process to form the preform having a central duct. The preform is heated to be partially collapsed to reduce the size of the central duct by using 3 collapsing passes. The partial collapsing passes are conducted through an externally traversing torch in the direction from the intake end of the preform to the exhaust end at decreasing torch traverse speeds. The traverse speeds for these three passes in an illustrative example are approximately 3.7 cm/min. for the first pass; approximately 2.9 cm/min. for

the second pass; and approximately 1.7 cm/min. for the third pass. The collapsing temperatures for the three passes are at about  $2150 \pm 100^\circ\text{C}$ . Oxygen flows through the central duct at a rate of at least 200 sccm during all three collapse passes. During the partial collapsing step, volatile dopants such as Ge and P diffuse out of the glass matrix. The central duct diameter is approximately 3 mm.

[0015] The region of glass with depleted dopants in the center portion of the preform is now etched. Etching is performed by flowing an etchant gas comprising a mixture of 6 sccm of  $\text{SF}_6$  and 194 sccm of  $\text{O}_2$  through the central duct while it is heated at a temperature of approximately  $1800^\circ\text{C}$ . The torch traverses in the direction from the preform's intake end to the exhaust end at a torch traverse speed of approximately 1.2 cm/min. to 21.0 cm/min., preferably 2.1 cm/min. during the etching step. The etch rate in this example is approximately  $0.01 \text{ cm}^3/\text{min}$ . At these etching conditions the central duct of the preform does not collapse. This lower temperature is important because it minimizes diffusion of fluorine into the glass and volatilization of the dopants. When the etching step of the invention is performed, the inner diameter of the tube can be as large as 5 mm.

[0016] After the etching step is completed, the central duct is closed at the exhaust end of the preform while 300 sccm of  $\text{O}_2$  flows to the central duct. This oxygen flow is reduced to zero as the central duct is closed. Finally, the preform is collapsed into a solid rod. The temperature is approximately  $2200 \pm 100^\circ\text{C}$  in the portion of the preform being collapsed. The central duct is closed along the length of the preform using a final collapse pass in the direction from the exhaust to the intake. The traverse speed for the final reverse collapse pass is about 1.9 cm/min. But a faster traverse speed may be used as long as the central duct closes in one reverse collapse pass. The entire collapsing and etching steps take about 3 or 5 hours depending on the tube diameter and the length of the tube.

#### Embodiment 2

[0017] A MCVD singlemode preform is manufactured from a 19X25 mm Heraeus Synthetic Quartz substrate tube. Glass layers are deposited as the core in the inner face of the tube using MCVD to form the preform having a central duct.

[0018] The preform is partially collapsed to reduce the size of the central duct by using 3 collapsing passes. The partial collapsing was conducted by an external traversing torch in the direction from the intake end of the preform to the exhaust end at decreasing torch traverse speeds. In an illustrative example, the traverse speeds respectively are about 2.6 cm/min. for the first pass; approximately 1.8 cm/min. for the second pass; and approximately 1.0 cm/min. for the third pass. The collapsing temperatures for the 3 passes are about  $2250 \pm 150^\circ\text{C}$ . Oxygen flows through the central duct at a rate of

at least 200 sccm during all three collapse passes. During the partial collapsing process, volatile dopants such as Ge and P diffuse out of the glass matrix. The resulting central duct diameter is approximately 1.5 mm.

[0019] The region of glass with depleted dopants in the center portion of the preform is etched. Etching is performed by flowing an etchant gas comprising a mixture of 6 sccm of  $\text{SF}_6$  and 194 sccm of  $\text{O}_2$  through the central duct while it is heated at a temperature of approximately  $1800^\circ\text{C}$ . The torch traverses in the direction from the preform's intake end to the exhaust end at a torch traverse speed of approximately 3.8 cm/min. during the etching step. The etch rate in this example is approximately  $0.01 \text{ cm}^3/\text{min}$ . At these etching conditions, the central duct of the preform does not collapse. This lower temperature is important because it minimizes diffusion of fluorine into the glass and volatilization of the dopants.

[0020] After the etching step is completed, the central duct is closed at the exhaust end of the preform while 300 sccm of  $\text{O}_2$  flows to the central duct. This oxygen flow is reduced to zero as the central duct is closed. Finally, the preform is collapsed into a solid rod. The central duct is closed along the length of the preform using a final collapse pass in the direction from the exhaust end to the intake end. The temperature is approximately  $2200 \pm 100^\circ\text{C}$  in the portion of the preform being collapsed. The traverse speed for the final reverse collapse pass is about 0.5 cm/min. although faster is better as long as the central duct closes in one reverse collapse pass. The entire collapsing and etching process take about 5-7 hours depending on the tube diameter and the length of the tube.

#### Embodiment 3

[0021] A graded index MCVD multimode preform using a Heraeus Synthetic Quartz substrate tube is manufactured. Glass layers for the core are deposited in the inner face of the tube using the MCVD process to form the preform having a central duct.

[0022] After deposition of the glass layers, the exhaust end of the preform is plugged with a device to prevent gases from exiting the preform. The preform is then partially collapsed to reduce the central duct by using 3 collapse passes. The partial collapsing passes are conducted by an external traversing torch in the direction from the end of the preform exhaust to the intake end at decreasing torch traverse speeds. The traverse speeds in an illustrative example are approximately 2.0 cm/min. for the first pass; approximately 2.0 cm/min. for the second pass; and approximately 1.4 cm/min. for the third pass. The collapsing temperatures for the 3 passes are about  $2150 \pm 100^\circ\text{C}$ . During the collapse process, a stagnant  $\text{O}_2$  environment is maintained within the tube. During the partial collapse step, volatile dopants such as Ge and P diffuse out of the glass matrix. The resulting central duct diameter is

approximately 3 mm. after this partial collapsing.

[0023] The region of glass with depleted dopants in the center portion of the preform is etched. Etching is performed by flowing an etchant gas comprising a mixture of 6 sccm of  $\text{SF}_6$  and 194 sccm of  $\text{O}_2$  while the preform is heated at approximately  $1800 \pm 150^\circ\text{C}$ . The device plugged at the exhaust end of the preform is removed to allow the gas to flow through the central duct tube. The torch traverses in the direction from the intake end to the exhaust end at a torch traverse speed of approximately 2.1 cm/min. during the etching step. At these etching conditions, the central duct does not collapse. This lower temperature is important because it minimizes diffusion of fluorine into the glass and volatilization of the dopants. The etch rate in this example is approximately  $0.01 \text{ cm}^3/\text{min}$ . Under the etching condition of this invention, the inner diameter of the central duct can be as large as 5 mm.

[0024] After the etching step is completed, the central duct is closed at the exhaust end of the preform while 300 sccm of  $\text{O}_2$  flows to the central duct. This oxygen flow is reduced to zero as the central duct is closed. Finally, the preform is collapsed into a solid rod. The central duct is closed along the length of the preform using a final collapse pass in the direction from the exhaust to the intake. The temperature is approximately  $2200 \pm 100^\circ\text{C}$  in the portion of the preform being collapsed. The traverse speed for the final reverse collapse pass is about 1.9 cm/min. But it may be even faster as long as the central duct closes in one collapse pass.

[0025] Fig. 2 shows a representation of the refractive index profile for a multimode fiber manufactured according to the present invention. It is evident that under the method of the invention, there is no dip in the center part of the preform in refractive index.

[0026] Fig. 3 shows a differential mode delay diagram for fibers produced by a prior art MCVD process and Fig. 4 shows a differential mode delay diagram for fibers produced by the present invention. These are actual data from fibers generated with and without the invention. The X-axis in the horizontal direction is time in nanoseconds. The Y-axis in the vertical direction is radius in microns with the bold line being the center of the fiber. The Z-axis which is perpendicular to the plane of the paper is the relative intensity. The dual pulse shown in Fig. 3 is an undesirable effect that may be detrimental in high-speed systems with restricted launch conditions. When the profile shape is optimized as shown in Fig. 2, this effect can be minimized.

[0027] By minimizing any defects in the center of the refractive index profile such as central dips, bumps and/or plateaus this effect can be minimized. These defects can be removed by this invention. Multimode fibers with no central dip have the additional advantage that the requirement of an offset patch cord is no longer required when the fiber is used in 1000 BASE-LX systems as described in the IEEE 802.3z standard.

[0028] The method described in this invention can

be used to minimize deviations in the central portion of the index profile. This process was developed for the MCVD process but is applicable to all glass optical waveguide preforms requiring the closure of a central duct including those fabricated using PCVD (plasma-enhanced chemical vapor deposition), and OVD (outside vapor deposition) processes. Control of the center portion of the profile allows one to manufacture fiber that is closer to theoretically calculated profiles. This added capability makes it easier to manufacture multimode and singlemode fibers with enhanced performance. One example of the utility of this invention is less pulse splitting in the center of a graded index multimode fibers that uses this invention. Another is better control of waveguide characteristics including mode field diameter and dispersion characteristics for singlemode fibers that use this invention.

#### Claims

1. A method of preparing a preform for an optical fiber, said method comprising the steps of:
  - heating a preform having a central duct at a first collapsing temperature to reduce the size of said central duct;
  - etching the surface of said central duct at a temperature lower than a minimum collapsing temperature of said preform; and
  - heating said preform at a second collapsing temperature to collapse said central duct of said preform.
2. The method of preparing a preform for an optical fiber according to claim 1, wherein said first collapsing temperature is lower than said second collapsing temperature.
3. The method of preparing a preform for an optical fiber according to claim 1 or claim 2, wherein said first heating step is performed with one end of said preform plugged with a device.
4. The method of preparing a preform for an optical fiber according to any of the preceding claims, wherein said etching step is performed by flowing an etchant gas through said central duct.
5. The method of preparing a preform for an optical fiber according to any of the preceding claims wherein said etchant gas comprises  $\text{SF}_6$ .
6. The method of preparing a preform for an optical fiber according to claim 5, wherein said etchant gas comprises a mixture of oxygen and  $\text{SF}_6$ .
7. The method of preparing a preform for an optical fiber according to claim 6, wherein said oxygen and

SF<sub>6</sub> have flow rates of 50-1500 sccm and 3.0-60.0 sccm respectively.

8. The method of preparing a preform for an optical fiber according to any of the preceding claims, wherein said etching step has an etching rate of 0.003-0.08 cm<sup>3</sup>/min. 5
9. The method of preparing a preform for an optical fiber according to any of the preceding claims, wherein said etching step is performed at a temperature about 200-400°C lower than said minimum collapsing temperature. 10
10. The method of preparing a preform for an optical fiber according to any of the preceding claims, wherein said etching step is performed at a temperature between 1600°C and 2000°C. 15
11. The method of preparing a preform for an optical fiber according to any of the preceding claims, wherein said etching step is performed by traversing a heating source external to said preform at a traverse speed of 1.2 cm/min. -21.0 cm/min. 20
12. The method of preparing a preform for an optical fiber according to any of the preceding claims, wherein said second heating step is performed with one end of said preform closed. 25

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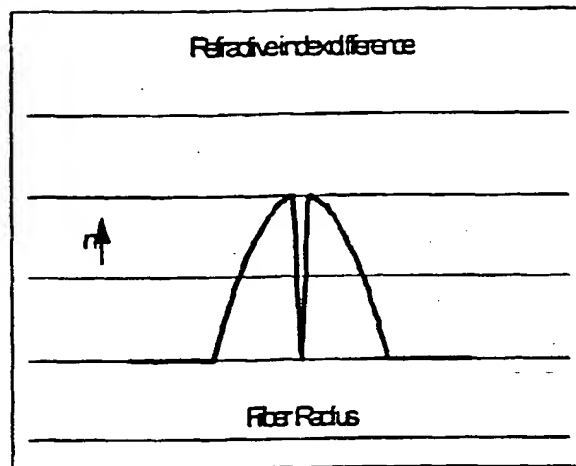


Fig. 1

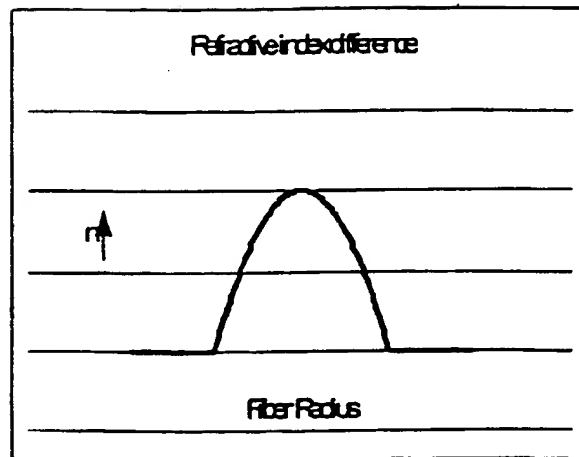


Fig 2



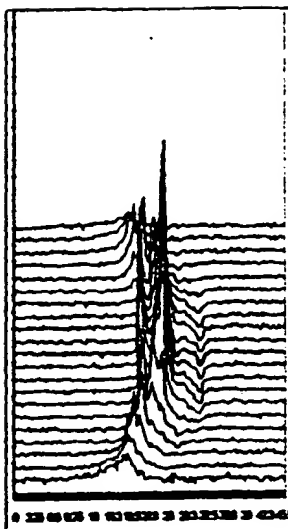


Fig 3

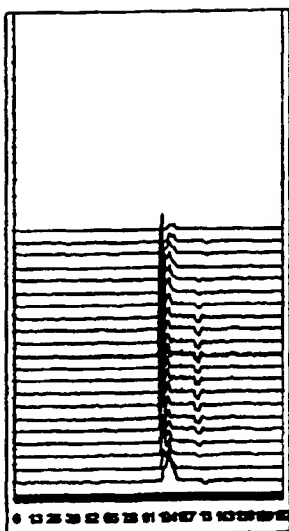


Fig 4.



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The present search report has been drawn up for all claims			
Place of search <b>THE HAGUE</b>		Date of completion of the search <b>9 May 2000</b>	Examiner <b>Stroud, J</b>
<div>CATEGORY OF CITED DOCUMENTS</div> <div> X : particularly relevant if taken alone  Y : particularly relevant if combined with another document of the same category  A : technological background  O : non-written disclosure  P : intermediate document </div> <div> T : theory or principle underlying the invention  E : earlier patent document, but published on, or after the filing date  D : document cited in the application  L : document cited for other reasons  a : member of the same patent family, corresponding document </div>			

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# EUROPEAN SEARCH REPORT

Application Number  
EP 00 30 1788

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Place of search <b>THE HAGUE</b>		Date of completion of the search <b>9 May 2000</b>	Examiner <b>Stroud, J</b>
<p><b>CATEGORY OF CITED DOCUMENTS</b></p> <p>X : particularly relevant if taken alone  Y : particularly relevant if combined with another document of the same category  A : technological background  O : non-written disclosure  P : intermediate document</p> <p>T : theory or principle underlying the invention  E : earlier patent document, but published on, or after the filing date  D : document cited in the application  L : document cited for other reasons  &amp; : member of the same patent family, corresponding document</p>			

EPO FORM 1503 08/02 (P/40201)

**ANNEX TO THE EUROPEAN SEARCH REPORT  
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EP 00 30 1788

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